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THE INFLUENCE OF THERMAL-MECHANICAL TREATMENTS ON THE FRACTURE --ETC(U)
SEP 78 L J BROUTMAN DAHC04-75-G-0169

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THE INFLUENCE OF THERMAL-MECHANICAL TREATMENTS ON THE FRACTURE OF POLYMERS

(9) FINAL REPORT. Jun 75 - 31 May 78

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September 25, 1978

(11) 25 Sep 78

U.S. ARMY RESEARCH OFFICE

(15) ~~Grants~~ DAHC 04-75-G-0169 ~~and~~ DAHC 04-77-G-0160

Illinois Institute of Technology
Chicago, Illinois 60616

(12) 8p.

400 234

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) The Influence of Thermal-Mechanical Treatments on the Fracture of Polymers		5. TYPE OF REPORT & PERIOD COVERED Final June 1975-May 31, 1978
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Lawrence J. Broutman		8. CONTRACT OR GRANT NUMBER(s) DAHC 04-75G-0169 DAHC 04-77G-0160
9. PERFORMING ORGANIZATION NAME AND ADDRESS Illinois Institute of Technology Department of Metallurgical and Materials Engineering Chicago, Illinois 60616		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office Post Office Box 12211 Research Triangle Park, NC 27709		12. REPORT DATE September 25, 1978
		13. NUMBER OF PAGES 6
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE NA

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

NA

18. SUPPLEMENTARY NOTES

The findings in this report are not to be construed as an official Department of the Army position, unless so designated by other authorized documents.

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

Residual Stress	Toughness
Impact Strength	Annealing
Polymer	Polycarbonate

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report summarizes the objectives of the research contract and lists the graduate students receiving financial support from this contract and the degrees which the students earned. Also, a list of publications resulting from this research is presented.

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Unclassified

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1.0 Summary of Research

One of the primary objectives of this research was to study the influence of residual stresses on the toughness and other physical properties of glassy amorphous polymers. Residual stresses have been produced by thermal quenching and by mechanical deformation at room temperature. Residual stresses and their influence on physical properties have never been seriously considered by investigators in the U.S. In the past, physical property changes in glassy amorphous polymers produced by thermal treatments have been explained by considering the possibilities of alterations to molecular conformations causing changes in thermal transitions, or by molecular ordering or crystallization, or by simply considering that quenched polymers have greater free volumes than well annealed polymers. One of the most fundamental accomplishments of this study has been to demonstrate that residual stresses can better account for physical property changes than can the above described mechanisms. Certainly, polymer investigators in the future must characterize the state of residual stress, prior to attempting to understand or explain observed phenomena. This is true not only for specimens subjected to thermal or mechanical deformations but also for molded or extruded specimens.

Specifically, the effect of thermal quenching on the properties of polycarbonate, acrylic and rubber modified polymers have been studied. It has been shown that when a pre-notched polycarbonate specimen is quenched in ice water from near its glass transition temperature, the measured Izod impact strength is improved. It was also demonstrated that other polymers such as PVC behave in an identical manner. It has been postulated that the compressive stresses created around the root of the notch suppress craze initiation and allow the polymer to yield prior to catastrophic crack initiation from the craze. This hypothesis is consistent with the results obtained from the cold working study. For polymers cold worked so as to primarily produce residual stresses without substantial orientation it appears that when a notch is placed into a compressive region, ductile failure occurs during impact as a result of craze suppression. It has also been shown that brittle polymers such as PMMA can be made to behave in a ductile manner when tested in flexure after thermal quenching. The flexure specimen can have permanent plastic deformation as a result of the surface compressive stresses which prevent brittle tensile failure prior to yielding in flexure.

Techniques for measuring residual stresses in polymers were also investigated. An ultrasonic stress analyzer was used for this purpose. The concept for determining stresses is to propagate a surface wave and measure the time delay as a function of stress level. Surface microhardness measurements were also attempted as a means of measuring residual stress levels. Both of the techniques proved impractical and unreliable for the determination of residual stresses.

The layer removal technique conventionally used for metals has been successfully adopted to measure the residual stress distribution through the thickness of a polymer sheet. Surface compressive stresses of approximately 3000 psi can be created in quenched polycarbonate and PMMA specimens. Tensile residual stresses as high as 2100 psi were present in 1/4 inch cold-rolled (multi-pass) polycarbonate at the surface and compressive residual stresses were present in the core. Similarly, maximum tensile residual surface stresses of 600 psi and 375 psi were present in cold rolled 0.275 inch PVC and 0.260 inch ABS, respectively. In all three polymers considered, the residual stresses at the surface decreased with increasing thickness reduction.

The influence of cold working on toughness improvement in glassy amorphous polymers, such as polycarbonate, ABS, PVC, etc. was studied. Cold working processes, namely, rolling (6in. diameter rolls), Steckel rolling (1/2 and 1 in. diameter rolls) and drawing (die angle as a parameter), were used to produce thickness reduction up to about 40 percent in flat strip specimens. The Izod impact strength, tensile properties, hardness, density, and the energy absorbed in three point bend tests for notched specimens were measured as a function of strip thickness reduction and process parameters. It was shown that the toughness enhancement in cold working was due to the residual stress state present in the cold worked polymer, as well as the degree of molecular orientation.

Residual stresses in 1/2 inch annealed polycarbonate after Steckel rolling (single pass) were investigated. At low roll reductions, the residual stresses were tensile at the surface and an Izod impact strength of 2 ft. lb./inch was noted, indicating brittle fracture. However, Izod impact specimens with the notch on the rolled surface showed ductile fracture. The analysis of the residual stress state shows that the presence of uniform compressive residual stresses at the notch tip across its width suppress the craze formation and its growth. This is also the first indication that polycarbonate specimens having a thickness of approximately 1/2 inch can fracture in a ductile mode in the Izod impact test.

Environmental stress crazing behavior in polycarbonate was studied as a function of the state of residual stress using carbon tetrachloride. Experiments show that the tensile stresses on the surface are detrimental to stress crazing resistance and good correlation has been made between residual stress level and crazing resistance.

Molecular orientation in cold-worked polymers was investigated by gradually heating the polymer in an Instron environmental chamber and noting the maximum force of retraction while keeping the specimen length constant. The stress is termed the orientation release stress. At low thickness reduction, the molecular orientation was almost non-existent. With the increase in thickness reduction, the molecular orientation increased. Data on cold-worked polycarbonate, PVC and ABS has been developed.

The orientation measurements showed that at low thickness reduction, the Izod impact strength improvement in cold-rolled polycarbonate was the result of the tensile residual stresses created on its surfaces and the resulting compressive stresses in the core. However, in PVC and ABS, the residual stress levels were not sufficient to enhance their toughness. The toughness increase in PVC at large thickness reduction was the result of molecular orientation, whereas in ABS the increase is linear with respect to roll thickness reduction. Orientation release stress measurements through the thickness showed that the degree of orientation in cold-rolled polycarbonate is greater at the surface than the core of the specimen.

Annealing experiments were conducted in order to relieve the residual stresses while maintaining the orientation. The properties of the annealed specimens were then determined. Residual stress measurements made on annealed specimens showed that a minimum tensile residual stress level of 500 psi is required to maintain a high level of toughness in notched Izod impact tests of 1/4 inch polycarbonate. With increase in annealing temperature (annealing time: 2 hours), an increase in roll reduction was required to maintain ductile behavior in the Izod impact test. This increase is attributed to loss of residual stresses due to annealing, while maintaining molecular orientation. Residual stresses and molecular orientation for low (approximately 2%) and high (approximately 35%) thickness reduction in cold-rolled polycarbonate were determined as a function of annealing time and temperature to underline the molecular aspects of toughness improvement. Long term (several hours) orientation release stress measurements at constant temperature were also made. These measurements showed that complete molecular orientation release at room temperature would take place (by extrapolation) in not less than 20 years for polycarbonate specimens with 20% minimum thickness reduction. Thickness growth after various annealing conditions was also noted.

Stress relaxation experiments were conducted at initial stress levels of 2100 psi and 4500 psi. Stress relaxation material constants for polycarbonate were empirically determined. The data predicts the stress relaxation behavior of residually stressed polycarbonate. The analysis showed that in residually stressed polycarbonate at low thickness reduction, a time period of 17 years would be required to relax the stresses to the stress level where brittle behavior is expected.

Finally, the effect of stress relaxation was considered for PVC and ABS polymers. For example, it was found that if the Izod impact strength of these polymers were determined within 24 hours of quenching a ductile failure was produced having a relatively large value of impact strength. At times greater than 24 hours, a brittle type failure was produced and this phenomena has been attributed to the relaxation of favorable residual stresses.

2.0 Graduate Students and Degrees Earned

The following graduate students contributed to the research accomplished during this grant period. The research of the students was used as the subject of their graduate theses. Those students receiving their degrees during the grant period are so indicated.

Student	Degree	Year
Paul So	MS in Metallurgical and Materials Engineering	June 1976
Mary Kong	MS (in progress) Metallurgical and Materials Engineering	
Bharat Thakkar	PhD in Mechanical Engineering	June 1977
Tat Wong	PhD (in progress) Metallurgical and Materials Engineering	
George Waring	MS (in progress) Metallurgical and Materials Engineering	

3.0 Publications

The following papers were published or are in press as a result of this research grant:

1. "The Effect of Residual Stress and Thermal-Mechanical Deformation on the Toughness and Crazing of Amorphous Polymers", in Proceedings of Third Conference on Deformation, Yield and Fracture of Polymers, Plastics and Rubber Institute, Apr. 1976 (L. J. Broutman and P. So).

2. "Residual Stresses in Polymers and their Effect on Mechanical Behavior" Proceedings of Annual Technical Conference Society of Plastics Engineers, Apr. 1976 (L. J. Broutman and P. So).

3. "Residual Stresses in Polycarbonate and their Effect on Mechanical Behavior", Poly. Eng. and Sci., 16, 12, 1976 (L. J. Broutman and P. So).

4. "Cold Working of Polycarbonates: 1. Residual Stresses and Impact Strength", Proceedings of Annual Technical Conference Society of Plastics Engineers, April 1978 (L. J. Broutman and B. Thakkar).

5. "Cold Working of Polycarbonates: 2. Residual Stress, Molecular Orientation and the Effect of Annealing", Poly. Eng. and Sci., (in press) (with B. Thakkar).

6. "Cold Working of Rubber Modified Polymers and the Effect of Residual Stress", Poly. Eng. Sci., (in press) (with B. Thakkar).

7. "Relaxation of Residual Stresses in Amorphous Polymers", Poly. Eng. and Sci., (in press) (with T. Wong).